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13. ABSTRACT *(Maximum 200 words)*

The near-surface regions of polycrystalline Si_3N_4 were modified by ion implantation and post-implantation annealing. Metallographically polished bars and disks 3 mm in diameter and ranging in thickness from 250 to 500 μm were implanted of co-implanted with Al^+, B^+ and N^+ to fluences up to 2×10^{16} ions/cm^2, using implantation energies up to 300 keV. Some of the implanted material was post-implantation annealed at temperatures in the neighborhood of 1100 °C. The indentation fracture toughness was found to increase by more than 15 % for certain combinations of fluence, implantation species and post-implantation annealing temperature.

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Surface Modification of Structural Ceramics by Ion Implantation and Post-Implantation Anneaing: Al_2O_3 and Si_3N_4

Final Report

Ion implantation experiments were performed on Si_3N_4 produced by Cercon Inc., Vista, CA, for the purpose of improving its fracture toughness. The material contained 6 wt. % Y_2O_3 and 3 wt. % Al_2O_3 as sintering aids, and was HIPped. Pieces measuring $3 \times 5 \times 5$ mm were cut from the bars provided and metallographically polished in preparation for the first ion implantations. In these experiments specimens of Si_3N_4 were implanted individually with B^+ and Al^+ or co-implantated with $B^+ + N^+$ and $Al^+ + N^+$. These choices were dictated primarily by the fact that B and Al are strong nitride formers. The ion implantations were performed at the Hughes Malibu Research Laboratories, using an implanter under the supervision of Dr. R. G. Wilson. The implantation conditions used are summarized in Table 1. The doses were small, but were conveniently attained within a few hours of implantation using the ion beam fluxes available. Post-implantation annealing was done for 24 h at 1000, 1100 and 1200 °C in a N_2 atmosphere, the purpose of which was to limit the extent of oxidation rather than nitriding the implanted specimens.

The apparent fracture toughness, K_c, was measured using the indentation-toughness method [1]. This method predicts that K_c is determined by the equation

$$K_c = \frac{\chi P}{c^{3/2}}, \tag{1}$$

where 2c is the length of the radial/median cracks that originate at the corners of the indentation under load P. The constant χ is obtained from the equation

$$\chi = \delta\left(\frac{E}{H}\right)^{1/2}, \tag{2}$$

where E is Young's modulus, H is the Vickers hardness and δ is another constant obtained from the equation [2]

$$\delta = \frac{\psi}{24(1-2\nu)(\sqrt{2}\pi\tan\phi)^{2/3}}, \tag{3}$$

where ν is Poisson's ratio (0.27 for Si_3N_4), 2ϕ is the apex angle of the Vickers indenter and ψ is another constant determined from the geometry of the crack. When the specimen is large compared to c, ψ takes on the value 1.2 [3-5], which we have used throughout.

Measurements were initially made using an indentation load of 3.5 kg, but at this value of P the indentation cracks that formed in the annealed specimens were difficult to observe, partly due to limited oxidation of the surface, hence subsequent specimens were indented using P = 7.35 kg (34.3 N). Within the limits of experimental error there were no significant variations of hardness with any combination of implantation and post-implantation annealing. This is undoubtedly not because implantation has no effect on hardness, but because the large loads used cause indentations that far exceed the ion ranges (Table 1).

The results of these experiments are summarized in Figs. 1 to 3. In general, implantation with Al^+ ions was more effective than implantation with B^+ ions in increasing K_c of the Si_3N_4, though the increase in K_c exceeded 15 % in only two cases (Al^+-as-implanted to a dose of 10^{16} and Al^+ annealed at 1100 °C after implanting to 2.5×10^{16} ions/cm^2). Co-implantation with N^+ was done to higher total doses, and noticeably reduces the values of K_c in the as-implanted condition. However, annealing the Al^+-implanted Si_3N_4 at 1000 °C increased K_c by about 18 % over that of unimplanted material, while the toughness of the B^+-implanted Si_3N_4 increased by about the same amount on annealing at 1100 °C.

A second series of implantation experiments was performed on metallographically polished bars and disks of Si_3N_4. The disks were 3 mm in diameter and ranged in thickness from 250 to 500 μm. The intent of these experiments was to perform controlled-flaw tests [6,7], which require indentation at specific loads prior to

testing, in preparation for testing in our miniaturized disk-bend testing apparatus [8]. The thinner disks were to be indented at lower loads, while the thicker ones were to be indented using larger loads (up to ~10 kg on our microhardness indentation machine). A summary of the irradiation conditions is presented in Table 2. Unfortunately, the grant period expired, the funds were exhausted, and it was not possible to complete the planned experiments on the mechanical behavior of the implanted disks.

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Table 1. Ion implantation conditions for the initial experiments.

Ion Species	Ion Energy (keV)	Fluence ($ions/cm^2$)	Range (nm)
Al	300	10^{15}	315
"	"	10^{16}	"
"	"	2.5×10^{16}	"
Al	300	2.5×10^{16}	315
+			
N	240	2.5×10^{16}	"
B	300	10^{15}	540
"	"	10^{16}	"
B	200	10^{16}	385
+			
N	300	10^{16}	"

Table 2. Ion implantation conditions for the Si_3N_4 disks and bars.

Specimen Type	Ion Species	Ion Energy (keV)	Fluence ($ions/cm^2$)
3×5 mm bar and 32 disks	Al + N	Al: 300 N: 225	2.5×10^{16} each
3×5 mm bar and 32 disks	B + Al	B: 200 N: 300	1.0×10^{16} each
3×5 mm bar and 32 disks	Al	300	2.5×10^{16}
16 disks	Al	300	10^{16}

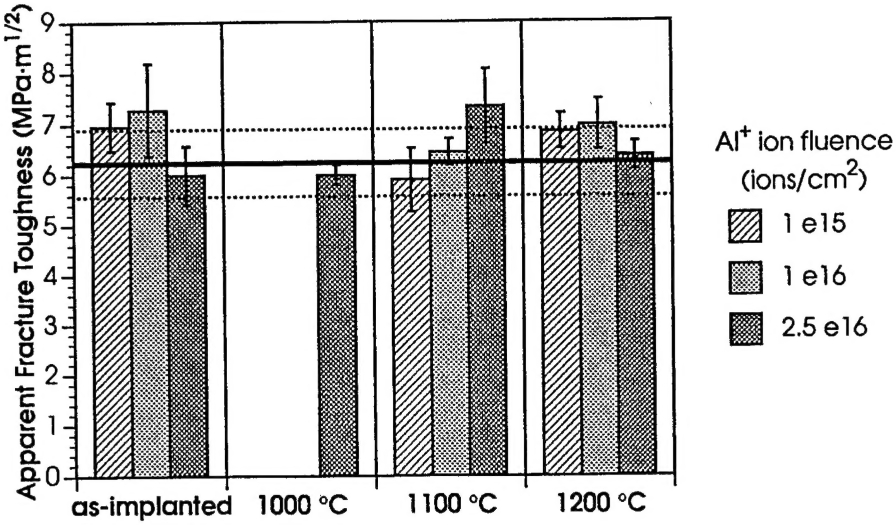

Figure 1. Apparent fracture toughness, K_c, of Si_3N_4 after implantation of Al^+ ions to the fluences indicated, and post-implantation annealing. The heavy horizontal line represents the average value of K_c of the as-received material, and the dashed lines the standard deviation. The Si_3N_4 implanted with Al^+ to the lower two doses was not annealed at 1000 °C.

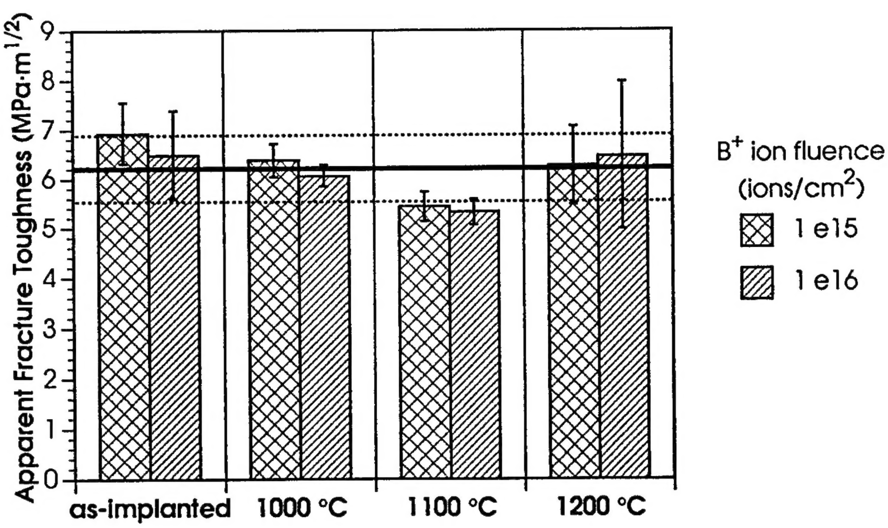

Figure 2. Apparent fracture toughness, K_c, of Si_3N_4 after implantation of B^+ ions to the fluences indicated, and post-implantation annealing.

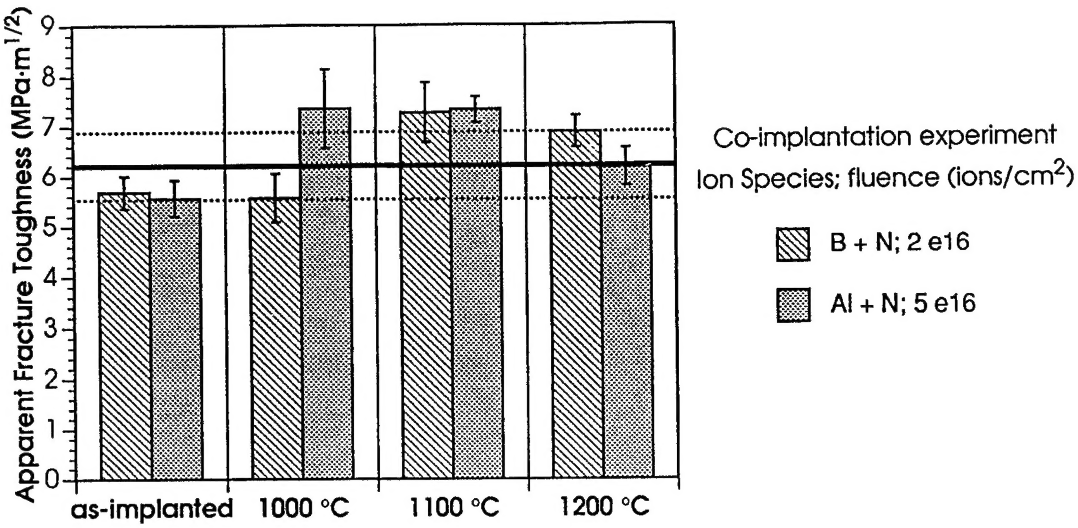

Figure 3. Apparent fracture toughness, K_C, of Si_3N_4 after co-implantation of Al^+ + N^+ and B^+ + N^+ ions to the fluences indicated, and post-implantation annealing.